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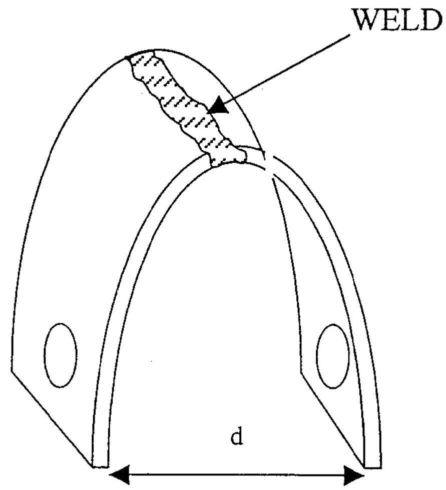

FIGURE 1: Schematic representation of the DUB U-bend specimens.

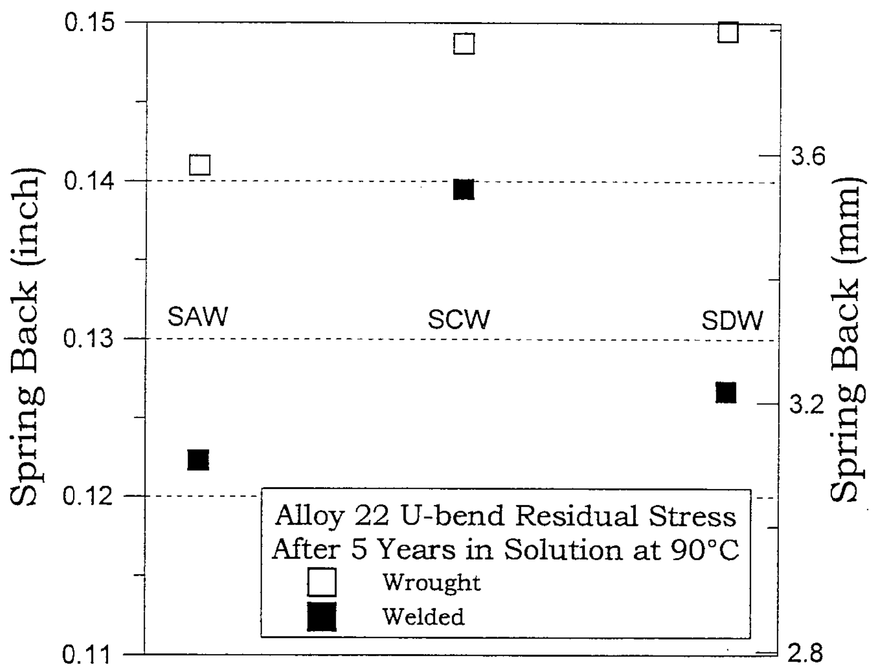

FIGURE 2: Spring-back after removing the bolt of U-bend specimens exposed for 5 years in corroding solutions.

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INFLUENCE OF ENVIRONMENTAL VARIABLES ON THE SUSCEPTIBILITY OF ALLOY 22 TO ENVIRONMENTALLY ASSISTED CRACKING

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ABSTRACT

In its current design, the high-level nuclear waste containers include an external layer of Alloy 22 (N06022). This material was selected to provide long-term corrosion resistance since if water enters in contact with the containers, they may undergo corrosion. The model for the degradation of the containers includes three modes of corrosion, namely general corrosion, localized corrosion and environmentally assisted cracking (EAC). The objective of the current research was to quantify the susceptibility of Alloy 22 to EAC in several environmental conditions with varying solution composition, temperature and electrochemical potential. The susceptibility to EAC was evaluated using constant deformation (deflection) U-bend specimens in both the wrought and welded conditions. Results show that after more than five years exposure in the vapor and liquid phases of alkaline (pH ~ 10) and acidic (pH ~ 3) multi-ionic environments at 60°C and 90°C, Alloy 22 was free from EAC.

Keywords: high-level nuclear waste, nickel-based alloy, N06022, environmentally assisted cracking, U-bend, welded specimens, slow strain rate test, acoustic emission, simulated acidified water (SAW), simulated concentrated water (SCW), simulated dilute water (SDW).

INTRODUCTION

The current design concept for the high-level nuclear waste containers in the USA is based on a metallic multi-barrier system. This design specifies an external layer of Alloy 22 (N06022) and an internal layer of type 316 stainless steel (S31603).[1,2] The main purpose of the internal barrier is to provide structural integrity and to contribute to the shielding of radiation. The main role of the external barrier is to provide protection against corrosion. Alloy 22 was selected for the external barrier due to its excellent resistance to general corrosion, localized corrosion and environmentally assisted cracking in a broad range of environments.[3-8] Alloy 22 is a nickel (Ni) based alloy that contains approximately 22% chromium (Cr), 13% molybdenum (Mo), 3% tungsten (W) and 3% iron (Fe). Because of its high Cr content, Alloy 22 remains passive in most industrial environments and therefore has an exceptionally low general corrosion rate. The combined presence of Cr, Mo and W imparts Alloy 22 with high resistance to localized corrosion such as pitting corrosion and crevice corrosion.

Mill annealed Alloy 22 is highly resistant to EAC or stress corrosion cracking (SCC) in acidic concentrated chloride solutions. [7-13] Dunn et al. did not find SCC when they tested Alloy 22 in 14 molal Cl^- (as $MgCl_2$) at 110°C and 9.1 molal LiCl at 95°C under controlled potential. [9-12] They used wedge opening loaded double cantilever beam (DCB) and compact tension (CT) specimens at stress intensities in the range 32 to 47 $MPa{\cdot}m^{1/2}$ for times as long a 52 weeks. [9-12] Rebak reported that Alloy 22 U-bend specimens did not suffer SCC when exposed to 45% $MgCl_2$ at 154°C for up to 6 weeks. [7] Estill et al. performed SSRT at a 1.6 x 10^{-6} s^{-1} strain rate at the corrosion potential (E_{corr}) in 4 M NaCl at 98°C, saturated $CaCl_2$ (>10 M Cl^-) at 120°C and 1% $PbCl_2$ at 95°C.[13] None of these specimens showed a loss of ductility or secondary cracking.[13]

Even though Alloy 22 is resistant to SCC in concentrated chloride solutions, it may be susceptible under other severe environmental conditions.[14-18] Andresen et al. tested the susceptibility of Alloy 22 to EAC at the corrosion potential (E_{corr}) in basic saturated water (BSW) at 110°C.[14] This BSW multi-ionic solution is a version of concentrated solutions that might be obtained after evaporative tests of Yucca Mountain ground waters.[19] Using the reversing DC potential drop technique, Andresen et al. reported a crack grow rate of 5 x 10^{-13} m/s in a 20% cold-worked specimen loaded to a stress intensity of 30 $MPa.m^{1/2}$. This EAC testing was carried out in air saturated BSW water of pH ~ 13. The testing conditions used by Andresen et al. were highly aggressive and, in spite of that, the measured crack growth rate was near the detection limit of the system.[14] Rebak et al. reported that Alloy 22 U-bend specimens suffered transgranular SCC when they were exposed for 336 h to aqueous solutions of 20% HF at 93°C and to its corresponding vapor phase.[15] The liquid phase was more aggressive than the vapor phase.[15] Pulvirenti et al. reported transgranular cracking in one out of four Alloy 22 U-bend specimen exposed for 15 days at 250°C in concentrated ground water contaminated with 0.5 % lead (Pb) and acidified to pH 0.5. [16-17] Estill et al. performed slow strain rate tests, cyclic loading tests and U-bend tests in large variety of environments (temperature, applied potential and solution composition).[13] They only reported SCC on MA Alloy 22 through SSRT in saturated concentrated water (SCW) at 73°C and at a potential of +0.3 to +0.4V [SSC].[13,18] When Alloy 22 was strained in SCW solution at +0.1 V [SSC], the sample did not suffer environmental assisted cracking (EAC or SCC).[18] The corrosion potential (E_{corr}) of Alloy 22 in SCW solution at 60°C and 90°C was in the order of 0 to +0.1 V [SSC].[20] That is, it is not expected that Alloy 22 would undergo SCC in SCW solution at the free corroding potential (E_{corr}).

The purpose of the present work was to evaluate the EAC characteristics of Alloy 22 in twelve different environmental conditions using constant deformation U-bend specimens. A few results are also presented regarding the used of acoustic emission to detect the onset of cracking during slow strain rate tests.

EXPERIMENTAL TECHNIQUE

There are several different techniques that can be used in the laboratory to study the susceptibility of alloys to EAC. The techniques can be grouped by the way the mechanical stress is applied to the testing specimen. In order to better simulate the likely field behavior, the samples that are used for laboratory testing should reproduce closely the field conditions. The only mechanical stresses that may be present in the containers at the Yucca Mountain Site would be residual stresses due to fabrication or possible rock fall impact. Therefore, the specimens chosen for laboratory testing were U-bend specimens, which also contained residual stresses due to permanent deformation.

The U-bend specimens were machined from sheet stock. The specimens were tested in the as-machined condition, which corresponded to a root mean square (RMS) roughness of 32 μ-inch. The specimens were degreased in acetone before testing. The U-bend specimens were prepared using ¾-inch (~19 mm) wide and 1/16-inch (~1.6 mm) thick strips according to ASTM G 30. The resulting specimen had a constant nominal separation between both legs, or ends, of 0.5 inch (~13 mm) secured by a bolt, which was electrically insulated from the specimen through ceramic zirconia washers. The total plastic deformation in the external outer fiber of Alloy 22 was approximately 12%. Single U-bends were produced using both wrought sheets and welded sheets. In the welded specimens, the weld was across the apex of the bend (Figure 1). The weld process was gas metal arc welding (GMAW) using filler metal and the seam had full penetration. Typical mechanical properties of MA sheet material are listed in Table 1. Table 2 lists the chemical composition of the sheet material and the filler metal used for the fabrication of the U-bend specimens.

A few tests were carried out using slow strain rate tests (SSRT). The specimens for this type of test were fabricated from plate stock. Typical mechanical properties of MA plate material are listed in Table 1. Table 2 shows the chemical composition of the SSRT specimens.

The testing electrolyte solutions for the U-bend were complex solutions containing several ionic species. The volume of the electrolytes was approximately 1000 liters. Table 3 shows the composition of the multi-component electrolyte solutions mentioned in this paper. Table 3 also shows the composition of the water from well J-13 at Yucca Mountain. The solutions used in this study are concentrated versions of J-13 water. The U-bend immersion tests were carried out at 60°C and 90°C. Approximately half of the specimens were exposed to the liquid phase of the solution and the other half to the vapor phase. The reported temperature corresponded to the liquid phase. The exposure time was approximately 5 years (the actual exposure time is given in Table 4). The electrolyte solutions were naturally aerated; that is, the solutions were not purged and the ingress of air above the solution level was not restricted. All tests were carried out under ambient pressure. The electrochemical potentials in this paper are reported in the saturated silver chloride scale [SSC]. At ambient temperature, the SSC scale is 199 mV more positive than the normal hydrogen electrode (NHE). After testing, the samples were evaluated using standard metallographic procedures such as optical and scanning electron microscopy.

EXPERIMENTAL RESULTS AND DISCUSSION

Constant Deformation Tests (U-bend Specimens)

The U-bend specimens were exposed to three different multi-ionic electrolyte solutions at the free corrosion potential (E_{corr}) for up to 5 years. Two of these electrolyte solutions (SCW and SDW) were alkaline of pH ~ 10 and one electrolyte (SAW) was acidic of pH ~ 3. Forty-nine (49) specimens were removed from six of the testing tanks, rinsed in de-ionized water and allowed to dry in the laboratory atmosphere. Table 4 lists the specimens by their label, by the vessel they were exposed to and by the length of time they were tested. The specimens were labeled starting with the letters DUA or DUB and followed by three sequential digits. The common letters D and U stand for Alloy 22 and U-bend, respectively. The letter A says that the specimen does not contain a weld or is fully wrought sheet and the letter B says that there is a weld seam across the apex of the specimen (Figure 1). In general, three specimens were examined for each temperature, solution composition and metallurgical condition.

The 49 specimens were first examined optically in a stereomicroscope using up to 100 times magnification. Six of these specimens were later disassembled (bolt removed), were examined in the scanning electron microscope and subsequently mounted for metallographic sectioning. Stereomicroscope studies showed that most of the specimens were completely featureless, that is, they appeared shiny metallic similar to the non-tested condition. Table 5 summarizes the observations for the different tested conditions. Most of the specimens had deposits of crystals (probably salts) from the electrolyte. The specimens that were exposed to the vapor phase had lower amount of deposits than the specimens exposed to the liquid phase. However, surface features suggest that the specimens exposed to the vapor phase had abundant condensation on them. The specimens that were tested at the higher temperature (90°C) in the liquid phase in general showed higher degree of discoloration than the specimens tested at 60°C. This may suggest that there was a larger interaction between the specimens and the environment at the higher temperature; however, most of the colors and deposits observed (Table 5) suggest that these were result of deposits from the environment rather than due to a reaction of the metal with the environment. Besides Alloy 22, the tanks listed in Tables 4 and 5 contained a large number of specimens made of other nickel alloys such as C-4, 825 and 625 and other materials such as titanium (grades 12, 7 and 16). The origin of the colors (e.g. golden/green/blue) is not yet known. The golden color was probably caused by the deposit of little crystals of this color on the surface. Some of these small crystals may be rich in iron. Studies of the scales and oxide films on the Alloy 22 specimens are currently in progress. An important observation form Table 5 is that none of the 49 examined specimens showed any indication of obvious corrosion and or cracking (EAC).

Of the 49 specimens listed in Table 4, six were selected for destructive analysis. The selected specimens were (1) wrought and welded exposed for 1916 days to SAW liquid at 90°C (DUA054 and DUB054), (2) wrought and welded exposed for 1869 days to SCW liquid at 90°C (DUA114 and DUB114) and wrought and welded exposed for 1813 days to SDW liquid at 90°C (DUA140 and DUB140). While assembled, the separation distance between both ends of the U-bend specimen (d in Figure 1) is kept constant by means of a bolt. This distance was measured for the six specimens mentioned above before and after removing the bolt. The measuring was done three times along the ¾-inch width of the sample. This test was performed to determine if the specimens had residual stresses in them, that is, to determine if the ends would spring back when unloaded. Thus, if the difference between the ends of the disassembled specimen was larger than the distance of the assembled specimen, there were residual stresses in the U-bend. The nominal separation in an assembled specimen should be 0.5 inch.

The average value of separation for all six specimens before disassembling was 0.5068 ± 0.008-inch. The average value of separation for all six specimens after disassembling was 0.6447 ± 0.011-inch. For each one of the six specimens, this difference was positive showing that all the specimens had residual stresses in them (Figure 2). Figure 2 also shows that this difference was higher for the wrought than for the welded specimens. This was not a result of the difference in environmental response between the wrought and the welded specimens but a result of the bending characteristics of these two types of samples during the original fabrication of the samples. Figure 2 shows that the spring back was approximately the same for the three solutions, the acidic SAW and the alkaline SCW and SDW. Results from Figure 2 suggest that Alloy 22 was resistant to environmentally induced cracking (EAC) or embrittlement even after five years immersion in the corroding solutions at 90°C.

The six specimens listed above were also studied in the scanning electron microscope. Some of the specimens showed micro cracking perpendicular to the mechanical residual stresses. These cracks in general were shallow (approximately 2 μm deep) and less than 0.1 mm in length. Micro cracks with different degree of depth and length were observed in most of the tested samples. Figure 3 shows a micro crack on the surface of specimen DUB140, which was exposed to SDW at 90°C. It is not clear that micro cracks such as the one in Figure 3 formed while in presence of the electrolyte solution. This crack could have been formed during the fabrication of the specimen. Figure 4 shows similar shallow micro cracks on the surface of specimen DUA156, which has never been tested (blank sample).

The same six specimens listed above were metallographically sectioned to determine if any shallow crack formed on the surface progressed deep into the bulk metal. Optical microscopy examinations of polished cross sections were carried out at up to 1000 times magnification. None of the six specimens showed any measurable crack length, which may have emanated from the surface. Figure 5 shows images of a cross section of a welded specimen exposed to liquid SDW at 90°C (DUB140). The minimum resolution of the microscope was in the order of 2 micrometer in length (Figure 5). That is, any crack shorter than 2 micrometers would not be discernible. Therefore, dividing this minimum measurable crack length by the nominal exposure time of 5 years, indicates that the described method of immersed U-bend specimens could have detected average crack growth rates of 1.3×10^{-14} m/s and larger (considering nil induction time). If a crack forms and grows at this given rate, it would take almost 50,000 years to propagate to a length of 20 mm.

Results from this testing show that wrought and welded Alloy 22 were highly resistant to EAC in multi-ionic solutions that could be representative of concentrated Yucca Mountain ground water. The tested specimens were free from cracking even after 5 years of immersion at the free corroding potential in such environments at 60°C and 90°C. The most typical values of E_{corr} for Alloy 22 in SCW and in SDW at 60°C and 90°C are in the vicinity of 0 V to +0.1 V [SSC] [20]. For SAW, E_{corr} could be higher, in the order of 0.35 V [SSC].[20]

Acoustic Emission During Slow Strain Rate Tests (SSRT)

Slow strain rate tests (SSRT) are highly aggressive since in this test the sample is deformed slowly and continuously until rupture. This type of test does not represent the conditions of the container, since the container may only have residual stresses. However, the SSRT is a useful fast technique to study environmental effects on the susceptibility of alloys to EAC. Specimens for SSRT were prepared from wrought mill annealed Alloy 22 plate (Table 2). Each specimen was cylindrical, approximately 7.25-inch (184 mm) long and 0.438-inch (11 mm) diameter. The useful gage of the specimens was 1-inch (25.4 mm) long and had a 0.1-inch (2.54 mm) diameter. Only the useful gage section was exposed to the electrolyte solution. Other areas of the specimens were covered with a protective coating. The slow strain rate tests were conducted at a constant deformation rate of 1.6 x 10^{-6} s^{-1}. It has been shown before that Alloy 22 exhibited EAC cracking after the samples were strained to rupture through SSRT in SCW at temperatures in the order of 80°C and at applied potentials of +0.3 to +0.4 V [SSC].[13,18] It was of interest to determine which level of stress was required for these EAC cracks to nucleate on the metal surface. Therefore, a couple of SSRT were conducted at two different applied potentials to detect noise generated by crack nucleation through acoustic emission. The tested conditions were as follows: (1) Sample ARC22-33 strained in SCW at 86°C at +0.4 V [SSC] and (2) Sample ARC22-29 strained in SCW at 88.5°C at +0.2 V [SSC]. The acoustic emission system was attached to the pull rod that was coupled to the strained sample. This system consisted on a sensor containing a piezoelectric transducer, which was able to detect elastic waves emitted by the strained specimen. Figure 6 shows a stress-strain curve for sample ARC22-33, which was deformed in SCW at 90°C and at an applied potential of +0.4 V [SSC]. Figure 6 also shows the number of times (hits or counts) that a waveform exceeded a certain pre-set threshold amplitude. Most of the hits happened for this specimen at an applied load higher than 700 lb. Since the original diameter of the sample gage was 0.1 inch, an applied load of 700 lb corresponded to an applied stress of 605 MPa. Therefore, data from Figure 6 suggests that cracking in Alloy 22 in the conditions given before started to nucleate at the high stress of 605 MPa and above under dynamic loading. According to data from Table 1, this corresponded to stress levels in the order of 80% of the ultimate tensile strength (UTS). Acoustic emission data for sample ARC22-33 showed that 2894 hits were recorded, while data for the ARC22-29 sample showed that only 262 hits were recorded. That is, the sample strained at +0.4 V [SSC] had a higher activity of acoustic emission than the sample strained at +0.2 V [SSC]. Previously published information shows that at an applied potential of +0.4 V a considerable amount of cracking was observed; however at +0.2 V only shallow fissuring was discernible on the specimen surface.[18] Identical results were observed for ARC22-29 and ASC22-33 specimens via scanning electron microscopy. That is, shallow barely noticeable cracking at +0.2 V [SSC] and more typical EAC at +0.4 V [SSC]. Therefore, it can be assumed that a correlation existed between the acoustic noise shown in Figure 6 and the amount of cracking observed after the testing. Once more, the acoustic emission analysis was useful to determine that cracking only initiated in the last stages of straining, when the applied stress reached levels near the UTS.

Final Observations

Environmentally assisted cracks did not form on rather smooth Alloy 22 wrought and welded specimens containing residual stresses and exposed at the free corroding potential to multi-ionic solutions at 60° and 90°C. Previous studies showed that it was possible to induce EAC on Alloy 22 using SSRT on anodically polarized specimens exposed to SCW solution.[18] Therefore, since it appears that Alloy 22 is not immune to EAC, it is planned to eliminate the residual stresses from the container surface before emplacement at the Yucca Mountain site. Thus, after fabrication, the container will be fully solution annealed. Once the waste is introduced into the container and the final closure welds are applied, the remaining residual stresses will the mitigated in situ, probably using laser peening. For example, this laser process may induce compressive stresses on the surface of the container to a depth of 3 mm.

CONCLUSIONS

(1) Mill annealed (MA) and welded Alloy 22 is highly resistant to environmentally assisted cracking (EAC) in multi-ionic solutions that could be representative of ground water at Yucca Mountain.

(2) U-bend samples exposed at E_{corr} for up to 5 years in SAW, SCW and SDW solutions at 60°C and 90°C were free from EAC.

(3) Alloy 22 was resistant to EAC at pH ~ 3 with E_{corr} of approximately +0.35 V [SSC] (SAW) and pH ~ 10 with E_{corr} of approximately +0.05 V [SSC] (SCW and SDW).

(4) Acoustic emission tests coupled to slow strain rate tests showed that EAC only nucleated in anodically polarized Alloy 22 at stress levels near the ultimate tensile stress.

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TABLE 1
TYPICAL MECHANICAL PROPERTIES OF PLATE AND SHEET ALLOY 22

Heat	Tensile Strength [UTS] (MPa)	Yield Stress [0.2%] (MPa)	Elongation to Rupture (%)	Hardness (RB)	ASTM Grain Size
Sheet – 2277-8-3203	824	412	62	92	5.5
Plate – 2277-8-3126	766	387	64.4	83	4

TABLE 2
CHEMICAL COMPOSITION OF THE STUDIED ALLOY 22 HEATS (Wt%)

Element	Single U-bend (Heat 2277-0-3264)	Weld Filler Metal (Heat 2277-4-3263)	SSRT (Heat 2277-6-3126)
C	0.004	0.002	0.004
Co	1.14	0.89	1.03
Cr	21.3	21.6	21.70
Fe	4.4	3.6	3.59
Mn	0.29	0.32	0.27
Mo	13.4	13.5	13.26
Ni	~56	~56	~56
P	0.01	0.009	0.006
S	<0.002	0.003	0.001
V	0.17	0.15	0.14
W	2.9	2.9	2.80

TABLE 3
CHEMICAL COMPOSITION OF THE ELECTROLYTE SOLUTIONS (mg/L)

Ion	SDW pH 10.1	SCW pH 10.3	SAW pH 2.8	J-13 Well Water pH 7.4
K^+	34	3400	3400	5.04
Na^+	409	40,900	40,900	45.8
Mg^{2+}	1	< 1	1000	2.01
Ca^{2+}	0.5	< 1	1000	13
F^-	14	1400	0	2.18
Cl^-	67	6700	24,250	7.14
NO_3^-	64	6400	23,000	8.78
SO_4^{2-}	167	16,700	38,600	18.4
HCO_3^-	947	70,000	0	128.9
SiO_3^{2-}	~ 40	~ 40	~ 40	61.1

TABLE 4
CONSTANT DEFORMATION (U-BEND) TESTS OF ALLOY 22.
LIST OF EXAMINED SPECIMENS

	SAW, 60°C	SAW, 90°C	SCW, 60°C	SCW, 90°C	SDW, 60°C	SDW, 90°C
Vessel	25	26	27	28	29	30
Wrought - Vapor Phase	DUA019, DUA020, DUA021	DUA049, DUA050, DUA051	DUA079, DUA080, DUA081	DUA109, DUA110, DUA111	DUA127	DUA139
Wrought - Liquid Phase	DUA022, DUA023, DUA024	DUA052, DUA054	DUA082, DUA083, DUA084	DUA112, DUA114	DUA128	DUA140
Welded - Vapor Phase	DUB019, DUB020, DUB021	DUB049, DUB050, DUB051	DUB079, DUB080, DUB081	DUB109, DUB110, DUB111	DUB127	DUB139
Welded - Liquid Phase	DUB022, DUB023, DUB024	DUB053, DUB054	DUB082, DUB083, DUB084	DUB113, DUB114	DUB128	DUB140
Date in	06Feb1997	21Feb1997	10Mar1997	10Apr1997	14Apr1997	05Jun1997
Date out	20May2002	21May2002	17May2002	22May2002	10May2002	22May2002
Exp. Time, days (h)	1930 (46,320 h)	1916 (45,984 h)	1895 (45,480 h)	1869 (44,856 h)	1853 (44,472 h)	1813 43,512 h)

TABLE 5
STEREOMICROSCOPE OBSERVATIONS OF THE TESTED U-BEND SPECIMENS

Conditions	Vapor Phase	Liquid Phase
Vessel 25 SAW, 60°C	Shiny metallic. Few isolated brown deposits. No corrosion or cracking	Shiny gray-green-blue. Brown deposits mostly in concave area. No corrosion or cracking
Vessel 26 SAW, 90°C	Shiny metallic or light gray. Brown deposits in concave area. No corrosion or cracking	Dark golden with green patches. Abundant brown deposits in concave area. No corrosion or cracking
Vessel 27 SCW, 60°C	Shiny and dull light gray with bluish and golden patches. Some white deposits. No corrosion or cracking	Shiny light golden. Some white deposits in concave area. No corrosion or cracking
Vessel 28 SCW, 90°C	Shiny dark gray and golden. Little white and green deposits in concave area. No corrosion or cracking	Sample covered by white salt-like deposits. Underneath deposits shiny light golden. No corrosion or cracking
Vessel 29 SDW, 60°C	Shiny light gray. Very little deposits. No corrosion or cracking	Shiny light gray. Little white deposits. No corrosion or cracking
Vessel 30 SDW, 90°C	Shiny light golden. No deposits. No corrosion or cracking	Shiny blue and golden. White deposits in concave area. No corrosion or cracking

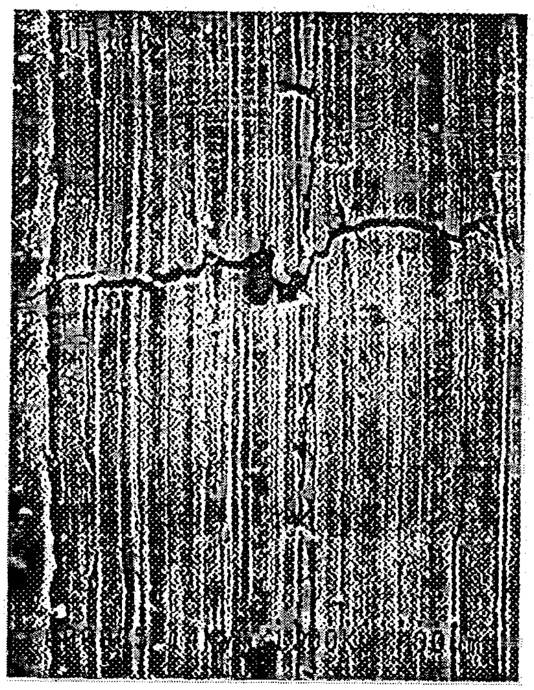
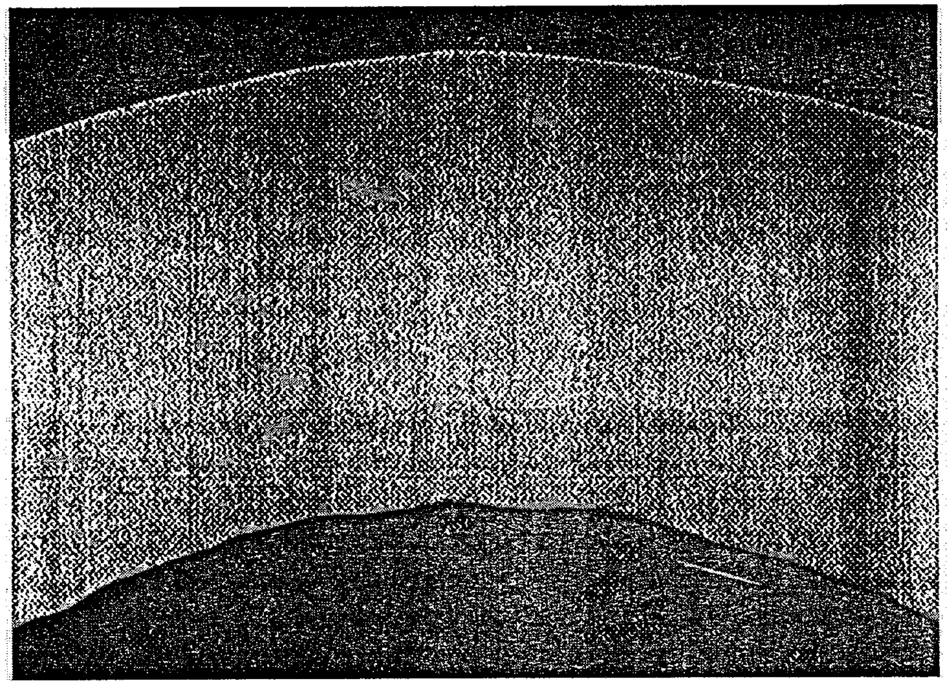
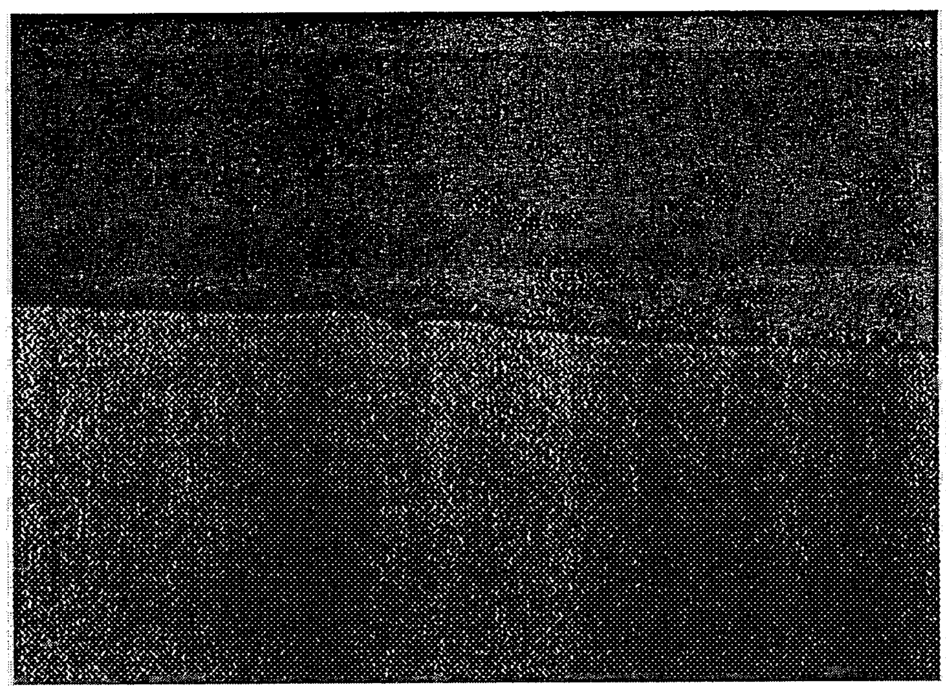


FIGURE 3: SEM Image of sample DUB140, exposed to SDW liquid at 90°C for 5 years. Shallow cracks are observed. Magnification X 1000.

FIGURE 4: SEM Image of sample DUA156, never tested (blank). Shallow cracks are observed. Magnification X 1000.



Magnification X 50



Magnification X1000

FIGURE 5: Optical micrographs of a cross section of sample DUB140. Sample is free of EAC. Surface notches are ~ 2 μm deep.

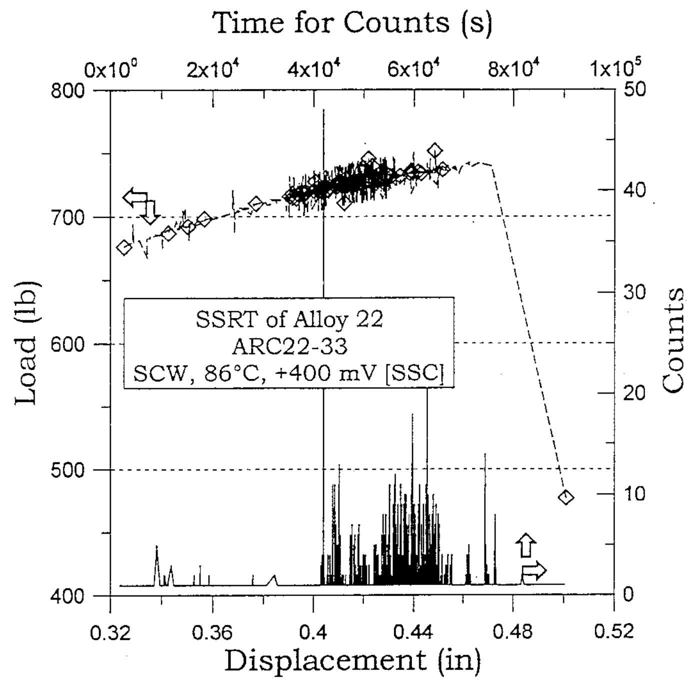


FIGURE 6: Final portion of the stress-strain curve for specimen ARC22-33 deformed in SCW at 90°C and at an applied potential of +0.4 V [SSC].
Acoustic emission data suggests that most of the cracking formed at a load level above 700 lb.